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(54) **Polycrystalline X-ray spectrometer.**

(57) A wavelength dispersive X-ray spectrometer is provided with a polycrystalline analyzer for analyzing characteristic spectra of a sample. The polycrystalline analyzer provides a multiple spectrum of characteristic lines which are separated by appropriate pulse height analysis. Each of these sets of characteristic lines of the elements of the sample are provided at different dispersion and wavelength ranges.

**EP 0 186 924 A2**

"Polycrystalline X-ray spectrometer."

The present invention is directed to a wavelength dispersive X-ray spectrometer allowing the separation in  $\theta$  space of intensities from a polychromatic beam of radiation excited from a sample consisting of many elements, wherein  
5 the individual intensities and therefore the individual elements can be measured. More particularly, the wavelength dispersive spectrometer utilizes a polycrystalline analyzing crystal for measuring a broad spectrum of  $\theta$  angles.

Wavelength dispersive spectrometers are commercially available in a broad range of devices. Primarily,  
10 such X-ray spectrometers are equipped with 3 to 6 analyzing crystals to allow a broad wavelength range to be measured. In particular, a wavelength range of from about 0.2 to 20 Angstroms can be measured.

In prior X-ray spectrometers, a number of single  
15 crystal analyzers have to be used in order to achieve measurements over a broad wavelength range. Principally, the spacings of individual single crystals were such that only a small part of the wavelength range could be achieved  
20 with an individual single crystal in the measurement of the spectrum, while maintaining optimum spectral dispersion. Consequently, a number of analyzing crystals were used with a dispersion in characteristic values in order to achieve measurement over a large wavelength spectrum.

The wavelength dispersive spectrometer provides  
25 separation in  $\theta$  space of intensities from a polychromatic beam of radiation excited from a sample consisting of many elements. Accordingly, by measurement of individual intensities, the elements and their concentration can be measured.  
30 ed. The separation in  $\theta$  space is achieved by allowing the beam of radiation from the specimen or sample to fall onto the surface of the single analyzing crystal. This crystal has been cleaved such that certain crystallographic planes

- are parallel to its surface. The measurable wavelength  $\lambda$  is related to the diffraction angle  $\theta$  by the expression  $n \lambda = 2d \sin \theta$  where  $n$  is an integer and  $d$  is the interplanar spacing of the single crystal used for diffraction.
- 5 The separation of lines in the spectrum is a function mainly of the  $d$  spacing of the crystal, and in fact, the angular separation  $d\theta$  of two wavelengths,  $\lambda_1$  and  $\lambda_2$ , of a wavelength difference  $d\lambda$ , is given by  $d\theta/d\lambda = n/(2d \cos \theta)$ .
- 10 Because of instrumental constraints, the range of  $\theta$  is limited to be between  $5^\circ$  and  $75^\circ$ . Accordingly, a large value of  $d$ , the interplanar spacing, must be selected for the measurement of a long wavelength, whereas the separation of lines improves with a decrease of  $d$  values. This
- 15 in turn requires that for the measurement of many wavelengths, i.e. many elements, several crystals must be employed. As noted above, traditional wavelength dispersive X-ray spectrometers are typically supplied with between 3 and 6 analyzing single crystals to allow the measurement
- 20 to be made over the wavelength range from about 0.2 to 20 Angstroms.

The presently claimed invention seeks to avoid the use of a multiple number of single crystals to obtain the wavelength spectra.

- 25 Accordingly, the analyzing crystal according to the present invention is a polycrystalline material and electronic analysis is provided of a spectrum.

The features and advantages of the present invention will be described in more detail, by way of example with reference to the drawing figures, in which:

Figure 1 is an example of the prior art;

Figure 2 illustrates the presently claimed invention;

Figure 3 illustrates the principle used in the prior art of Figure 1; and

Figure 4 illustrates the operation of the present invention.

A wavelength dispersive spectrometer of the prior

art using single crystals for wavelength measurements of a material to determine characteristic elements of the material is seen in Figure 1. An X-ray tube 1 directs an X-ray beam onto a characteristic sample 2. Each element in the sample emits a characteristic X-ray line series of radiation onto an analyzing crystal 3, which is one of a number of single crystals. Each single crystal disperses radiation from the elements according to the d spacing of that particular single crystal. In the interest of simplicity, only three characteristic lines 11, 12 and 13 are shown in the figure. Each of these characteristic lines represents individual wavelengths to be measured, thereby identifying the material.

According to the present invention, a polycrystalline analyzing crystal 5 is utilized, as seen in Figure 2, instead of the single crystal structure of Figure 1. The polycrystalline arrangement 5 enables each set of planes in the crystal lattice to diffract with the characteristic lines.

The polycrystalline structure 5 can be made by a single crystal which has been ground up and pelletized to the dimensions of an analyzing crystal, or it can be a polycrystalline mixture of two or more materials chosen to give the required characteristics.

Again, in the interest of simplicity, only two sets of characteristic lines are shown in Figure 2, as indicated by the two sets of lines 11, 12 and 13, as well as 11', 12' and 13'.

Because the dispersion of the spectrometer is a function of the d spacing causing diffraction, the dispersion of these two sets of lines is different. The result of using the polycrystalline analyzing crystal 5 is to produce multiple sets of line spectra, superimposed on top of each other. Each one of these spectra is characteristic of the elements in the sample, but each one has a different dispersion and wavelength range. By use of a pulse height selection circuit 20-22, seen in Figure 2, for example, calibrated automatically with angular calibration of the

attenuation, it is possible to tune the scaling and counting electronics such that just one of the spectral series is selected.

That is, by utilizing a proportional counter 20 feeding a pulse height selector 21, the different spectral series can be selected. This selection may be further seen by reference to Figures 3 and 4. The different spectral series are analyzed in a computer 22.

Figure 3 illustrates the principle used in the prior state of the art. Since the analyzing crystal, such as 3 in Figure 1, is scanned with a goniometer structure through a range of diffraction angles  $2\theta$ , an intensity/angle diagram is generated as shown in Figure 3. Since the diffraction angle is inversely related to the energy of the diffracting photons, as the value of  $2\theta$  increases, the energy of the radiation being diffracted decreases. Accordingly, the spectrum can also be considered as an intensity/energy diagram.

A proportional detector, like 20 in Figure 2, is used to convert the energy of the diffracted X-ray photons to individual voltage pulses with the pulse rate from the proportional detector being a measure of the photon rate entering the detector. In the technique of pulse height selection, a fixed voltage level acceptance window is employed in a pulse height selector, such as 21 in Figure 2, to reject all extraneous and noise pulses. However, since the magnitude of each output pulse from the detector is proportional to the photon energy, the size of the individual voltage pulses being diffracted also decreases with goniometer angle, and it is necessary to attenuate the voltage pulses leaving the detector so that they will fall within the preselected acceptance window of the pulse height selector. This attenuation is performed using a linear resistor which is coupled to the goniometer on which the analyzing crystal is mounted giving a sinusoidal attenuation factor. The system is calibrated by adjusting the gain of the proportional detector 20 by varying in turn the high voltage on the detector. The acceptance by

the pulse height selector 21 is indicated at the top of Figure 3.

In the present invention, the selection of a given spectral series is made by simply adjusting the gain of the counting system. Thus, to accept the series of lines 11, 12 and 13, for example, a first gain setting is chosen. To accept a second series of lines 11', 12' and 13', for example, another gain setting is chosen. These gain settings are typically controlled by a microprocessor, such as 22 in Figure 2, so that the selection of one line series or another is completely automated. In Figure 4, the spectrum is shown as it would be acquired according to the present invention. The acceptance by the pulse height selector 21 for the two mentioned different gain settings is shown at the top of the Figure.

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(CLAIMS)

1. In an X-ray spectrometer including a source of X-rays, a sample receiving X-rays from said source, said sample emitting characteristic radiation, analyzing means receiving said characteristic radiation for producing  
5 reflected radiation, and detector means receiving said reflected radiation for detecting concentrations of said sample, the improvement comprising said analyzing means being a polycrystalline material.

2. An X-ray spectrometer according to Claim 1,  
10 wherein said detector means includes a programmable pulse height selector circuit for providing a plurality of acceptance windows, each being sufficiently narrow to preselect a single discrete diffractogram at a time.

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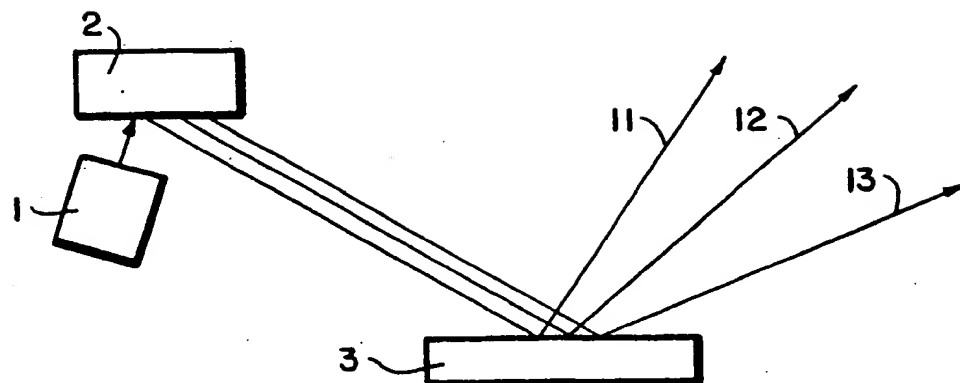


FIG. 1

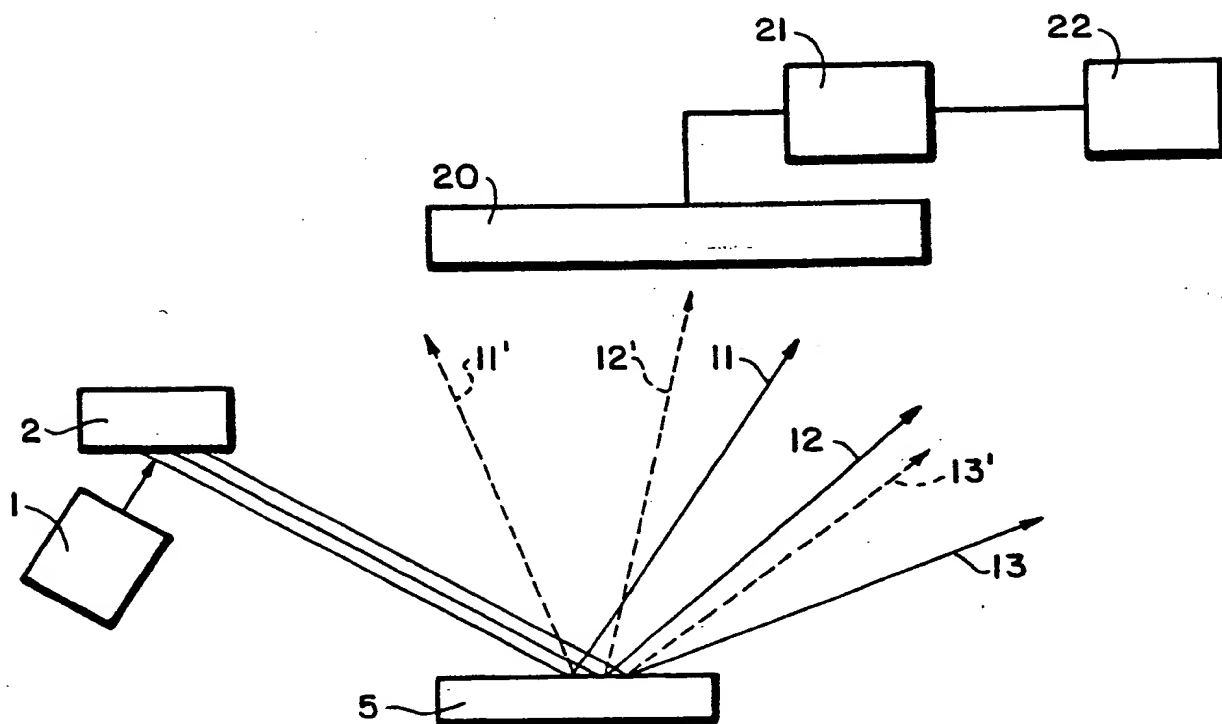


FIG. 2

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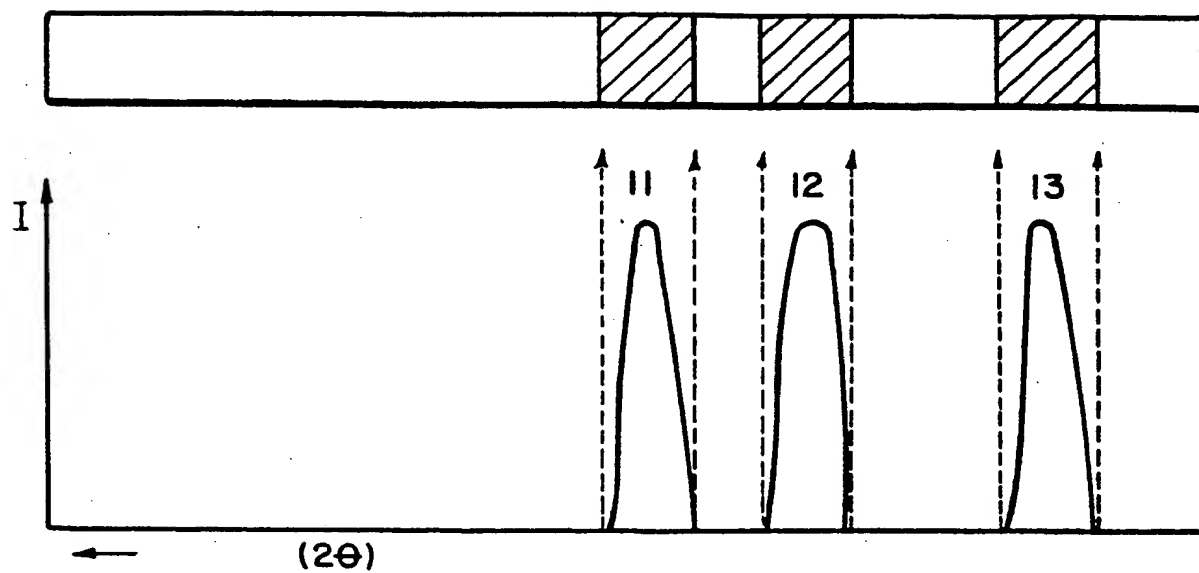


FIG.3

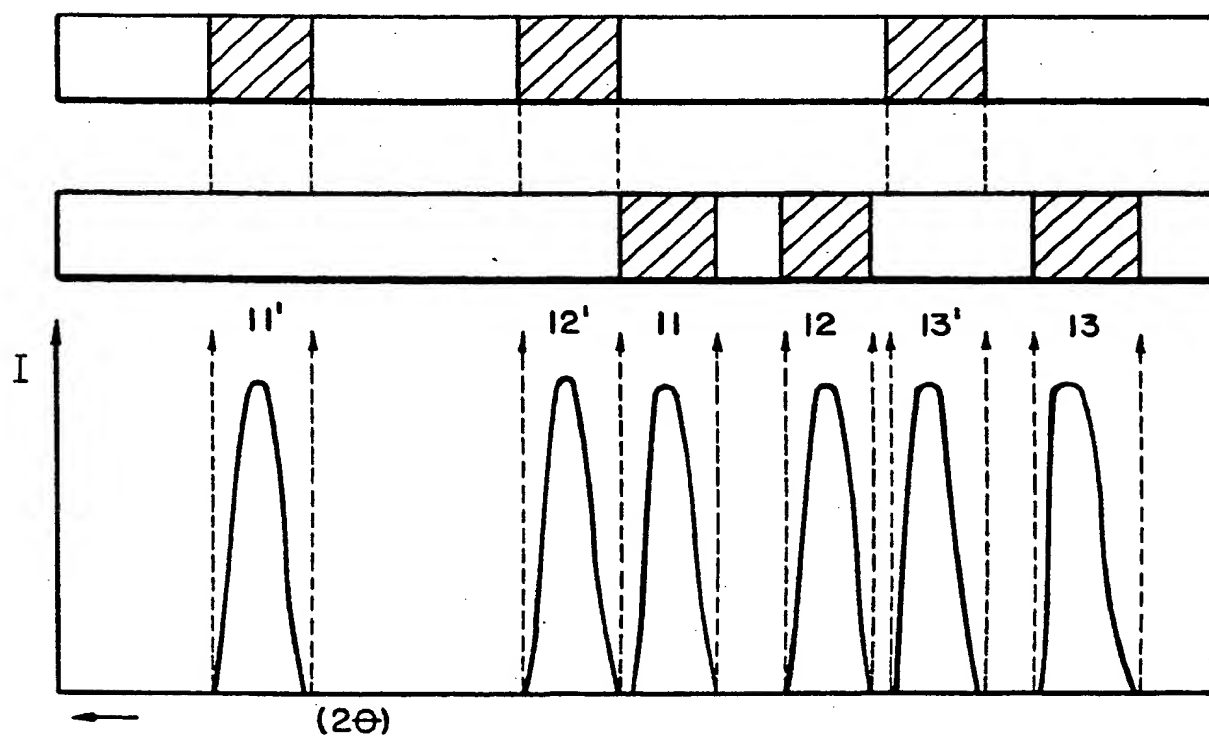


FIG.4

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54 X-ray diffraction inspection system and method.

57 An inspection system and method for detecting the presence of selected crystalline materials, such as explosives or drugs, utilizing an x-ray source (10) and a collimated array of detectors (32) to sense radiation scattered by suitcases (14) being inspected. A signal processing system (Fig. 4) comprising a photon energy detector (40), an energy dispersive spectrum generator (42), a peak detector algorithm (44) and an e.g. explosives spectrum comparator (46) compares the measured signal with selected spectra (Fig. 3a, b or c) to determine whether specific materials are present within the inspected suitcase.

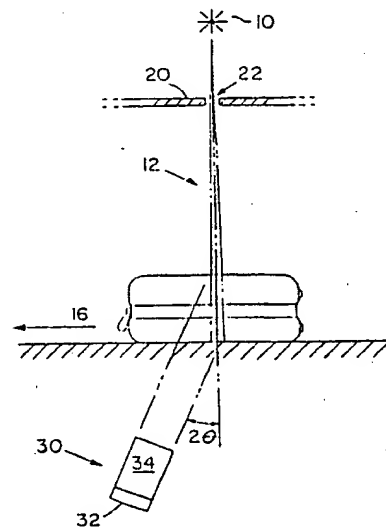


Fig. 2

## Description

## X-RAY DIFFRACTION INSPECTION SYSTEM AND METHOD

The invention relates generally to the field of radiographic detection systems, and more particularly, although not exclusively, to coherent x-ray scattering systems used to inspect objects to detect the presence of explosive materials.

Numerous systems have been developed for the inspection of bags, suitcases and briefcases etc., hereinafter more generally referred to as "parcels", that are used to screen travellers at airports or to secure other types of installations. Of particular concern in the development of such systems has been the detection of concealed weapons, explosives or drugs whose transport is restricted. Typically, standard x-ray equipment has been used to scan for metal objects where the outline of the displayed items is visually inspected to determine the presence of the objects of concern. These systems use a conveyor to transport the item to be inspected into and out of a chamber positioned between an x-ray source and a fluorescent or scintillation screen that detects the transmitted radiation. These inspection systems must be safe for the operators, and must not damage items such as photographic film that are often contained in inspected baggage.

Unfortunately, many kinds of materials that may pose a danger may not conform to any easily identifiable shape and are not visually detectable in the systems currently in use. In particular, many types of explosive materials in use can be molded into any shape and are not detectable by standard x-ray equipment. Thus, there is a need for a more accurate and dependable system for detecting these materials.

An inspection device of the present invention utilizes an x-ray radiation source to scan objects to determine whether certain crystalline substances are contained therein. Most explosives and many other dangerous or illegally transported substances have a crystalline structure.

Crystalline materials are known to scatter radiation when illuminated by an x-ray source of suitable energy and spectral content. Coherent elastic scattering of radiation occurs from lattices of spacing "d" within the crystalline material which satisfy the equation  $\lambda = 2d \sin \theta$  where  $\lambda$  is the wavelength and  $\theta$  is the angle of diffraction of the scattered radiation away from an axis through the source and the area being scanned. Thus, elastic scattered will occur at a few select wavelengths whereas inelastic scattering by the atoms of the material will occur continuously across a spectrum of wavelengths.

A detector positioned at the appropriate angle relative to the source and scanned object is fitted with a collimator that permits only those x-rays diffracted at that angle to reach the detector. The detector is preferably comprised of a photon spectrometer. Such spectrometers provide both intensity and spectral composition of the detected radiation and are typically either solid state germa-

nium or silicon planar arrays operated at cryogenic temperatures.

Each scattered photon is detected individually and a wavelength spectrum of intensities, displayed as an energy dispersive spectrum, is generated by a signal processing circuit.

A peak comparator algorithm incorporated into a data processing system is applied to the spectral output to determine the presence of sharp peaks within the spectrum. By comparing the peaks from the processed spectrum with the spectrum of crystalline substances sought to be detected the presence of those materials within the object is determined with great accuracy.

The present invention also provides a method of inspecting parcels to detect the presence of selected crystalline materials in the presence of other crystalline and noncrystalline materials comprising generating x-ray radiation from a source; conveying an object to be inspected past the source to irradiate the object with the radiation; and detecting radiation scattered by the object at a predetermined angle to detect the presence of a selected crystalline material on or within the object.

Some ways of carrying out the present invention will now be described in detail by way of example with reference to drawings which show one specific embodiment.

In the drawings:

FIG. 1 illustrates a front schematic view, of a parcel inspection device of the present invention;

FIG. 2 shows a side view of the device shown in Figure 1;

FIGS. 3a, 3b and 3c are illustrative graphical representations of diffraction spectra of particular explosive materials; and

FIG. 4 is a schematic diagram of the processing system of the device.

With reference now to the accompanying drawings, the inspection device is comprised of energy dispersive X-ray detectors arranged to measure the coherent elastic scattering of x-ray photons from the lattices of crystalline materials and in particular of crystalline explosives and of narcotic or hallucinogenic drugs. Nearly all of the explosives of interest comprise crystalline powders. For example, the plastic explosives are manufactured from crystalline powders of cyclotrimethylene-trinitramine (RDX), cyclotetramethylene-tetranitramine (HMX) and pentaerythritol-tetranitrate (PETN), and are compounded into a putty with minor amounts of organic binders. Each of the explosives which are to be detected provides a unique diffraction pattern when irradiated with x-rays such that each may be rapidly identified. The only notable exceptions are the nitro-glycerine-based dynamites. Fortunately these explosives are easy to detect by their vapor emissions and the two detection systems can be combined into a single instrument.

The device has sufficient speed of response to

detect explosives in bags which are conveyed through the detection zone in a few seconds. X-rays from source 10 are arranged in a beam 12 having a fan pattern to irradiate bags 14 which are conveyed along conveyor 16 through the beam 12. The beam 12 comprises an x-ray continuum whose range of photon energies is sufficient to penetrate large checked bags. The beam 12 is produced by collimation of the single x-ray source 10 of constant potential with slit collimator 20.

Photons scattered elastically from the crystalline lattice of explosives interferes coherently depending on the lattice structure, the frequency and angle of scatter. At a fixed angle of scatter, scattered rays comprise a continuum from non-crystalline materials and photons which are coherently scattered from the various 'd' spacings of the crystals. In a typical two dimensional lattice there are three major 'd' spacings which allow coherent diffraction. There are other lattice spacings in the third dimension so that for randomly oriented crystals there are sufficient 'd' spacings to provide a unique set, but not so many that they cause an overly cluttered diffraction spectrum.

The x-rays scattered in a beam of fixed angle defined by the detector collimator 34 comprises a continuum with superimposed intensity peaks at wavelengths  $\lambda_{1,n}$ .

The detection system 30 measures the intensity of scattered light in intervals of wavelengths over a wide range of photon energies but at a fixed angle  $2\theta$  of scatter. This provides a unique fingerprint for each type of explosive.

An array of individual detectors 32 is arranged across the full width of a conveyor system irradiated by an x-ray fan beam 12. This permits scanning of the whole volume of a bag 14.

The detector 32 employed in the system is a planar germanium photon spectrometer. These detectors operate as photon counters by detecting the liberation of free electrons by an x-ray photon in a germanium semiconductor. The total charge liberated by each x-ray photon is indicative of the photon energy thus allowing an intensity spectrum to be generated over a range of x-ray wavelengths. Typical resolution (full-width-half-maximum) provided by commercial detectors is 500eV at 120 KeV photon energy. The detector for this embodiment has a 100% detection efficiency over a range of photon energy from 5 KeV to 120 KeV.

The system is based on the detection of presence of two or three peaks in the spectrum. It is unlikely that the detection of these peaks will be interfered with by other crystalline materials. For example, the only crystalline material normally encountered in detectable quantities in 99% of airline bags are the metals aluminum, steel, and copper. The sensitivity of the inspection system is proportional to the total amount of crystalline material in a bag, so for small amounts of these metals only a low intensity of scatter is expected and this information is in parts of the spectrum which are distinct from the explosives or drugs of interest. Large amounts of metal can possibly stop 120 KeV photons and this condition can be detected by the attenuation of the trans-

mitted beam in a similar way to standard airport baggage x-rays, but no image would be generated. This condition is not expected to exist in more than a fraction of a percent of airline bags, and of course attempts to conceal the explosive by this method results in a rejection because of too high a metallic content.

The principle of detection is to send a "white" x-ray beam 12, containing a range of wavelengths, through the specimen and look for diffracted radiation at a fixed angle  $2\theta$ , with a detector system 30 that simultaneously measures intensity and wavelength. The advantages of energy dispersive x-ray diffraction over the more common monochromatic powder technique are two fold: the large sensitive volume (and therefore freedom from the necessity of aligning the sample precisely), and the fact that an entire spectrum may be collected in parallel.

In a crystal, there are regular planes of atoms separated by well-defined distances  $d_{1,n}$ . X-rays of wavelength  $\lambda$  may be scattered by these planes through a total angle  $2\theta$  if they meet the diffraction condition

$$\lambda = 2 d \sin\theta$$

The set of "d" spacings in a particular material, along with the associated intensities of diffraction, provide a fingerprint of the material. The system simply recognizes a stored pattern as an indication that a given material is present. Figures 3a, 3b and 3c show examples of spectra of three particular explosives, namely trinitrotolumine (TNT), so called "C4" plastic explosive, and "Flex-x", respectively. These spectra show peaks which distinctly identify these explosives.

Figure 4 illustrates the processing system in greater detail. The collimated germanium detector system 30 is used with an x-ray generator 24 capable of operating at up to 160 KV. A narrow beam 12 of x-rays is generated which irradiates a container 14 holding an explosive. The photons scattered through a fixed angle of  $2\theta$  were detected and all other scatter angles were precluded by a narrow aperture collimator 34 (Fig. 2). Thus spectrum of x-rays emerges from the source 10, but only those scattered at or near an angle of  $2\theta$  are seen by the detector.

A detector supply 56 provides the high voltage and temperature control system necessary to operate the germanium detector array 30. For each x-ray photon incident upon the detector, an electrical pulse is produced proportional to the energy, and therefore inversely proportional to the wavelength.

A photon energy detector 40 operates as a charge integrator which produces a digitized signal which is then processed by an energy dispersive spectrum generator 42. The generator 42 actually counts the number of photons within each of a number of energy intervals for each detector 32. For example, there are 140 separate 500eV intervals in an energy range between 50 KeV and 120 KeV. Thus each detector 32 generates a spectrum displaying the number of photons counted for an inspected item as a function of photon energy.

The processing sequence is then split into two

paths. The outputs from each individual spectrum generator 42 are processed by a peak detector algorithms 44 along one of these paths. Each algorithm 44 identifies those peaks within the spectrum from spectrum generator 42 which can be distinguished from the background spectra. The identified peaks are then isolated and quantified and output as a peak spectrum. The spectrum generated by the algorithm 44 for the individual detectors is then compared using the explosives spectrum comparator 46 with a number of predetermined spectra similar to those shown in Figures 3a, b and c. A sufficiently close match between the peak spectrum and one or more of the stored spectra will then identify the materials detected and display the result 54.

A second processing path that can be performed simultaneously with the above processing sequence takes the output from each spectrum generator 42 and sums all of the spectra at 48. The summed spectra is then analyzed with the peak detector algorithm 50 and compared to the stored spectra in the explosives spectrum comparator 52 to determine an aggregate result for the entire parcel being inspected. Thus in situations where the signal to noise ratio is too small for the individual detectors to properly identify the contents of the parcel, the summing of all the spectra will increase the ratio for materials distributed throughout the parcel sufficiently to properly identify the contents.

It is also possible to gate the detection and processing system at predetermined time intervals during the scanning of each particular bag to further increase the sensitivity of the system.

The sensitivity of the diffraction technique depends upon the total signal collected, which, in turn, is a product of the x-ray flux density  $F$ , the illuminated volume  $V_i$ , the time that the sample is in the beam, and the detector aperture seen from the sample. Consider the case of a bag containing a total volume  $V_o$  of explosive material, moving at a certain speed through the x-ray fan beam of width  $z$ . For simplicity assume the explosive is a rectangular block of dimensions (a)(b)(c), moving along the "a" direction.

Then, while it is in the beam the illuminated volume is:

$$V_i = (b)(c)(z),$$

and the time that it is in the beam is  $a / s$ . Consequently,

$$V_i t = V_o z/s,$$

and the overall detection sensitivity from the summed spectra for the whole bag is proportional to  $F V_o z/s$ .

It is important to emphasize that this result is independent of the shape and orientation of the explosive material within the bag.

The total radiation dose received by the inspected bag is equal to the product of the x-ray flux density and the time that one point is within the beam. The radiation dose is proportional to  $(F)(z)/s$ . Note that the x-ray beam properties and the bag speed appear identical in both expressions for the sensitivity and dose. This implies that, for a given acceptable radiation dose, the sensitivity is independent of

inspection speed. One can increase the x-ray power and bag speed proportionally without affecting either dose or sensitivity.

The collimator slit 22 adjacent to the generator 10 provides a variable fan beam 12 which is scattered and detected with a number of detectors fitted with a second set of collimators 34. The detector collimators 34 will be arranged to pass x-rays which are scattered through a narrow angle, 29, from samples mounted in the beam or held in a suitcase in the beam as shown. The angular aperture of the collimators 34 is on the order of  $0.03^\circ$ . Thus only those rays scattered through an angle of  $2 \pm .03^\circ$  are allowed into the detectors 32.

The detector 32 has a detection area which is capable of detecting scattered rays from a cross section of baggage for the full depth of a large suitcase and a strip of about 20 to 30mm wide across the bag. Several such detectors 32 are arranged across the conveyor having identical scatter angle and detect scattered rays from all of the bag volume.

## Claims

1. A parcel inspection device for the detection of selected crystalline materials in the presence of other crystalline and noncrystalline materials comprising:

an x-ray radiation source to irradiate an object to be inspected such that the radiation is projected along an axis through the source and object;

a conveyor for transporting objects through the projected radiation;

a detector positioned to receive radiation scattered by the object at an angle relative to the source-object axis; and

an analyzer to determine the presence of a predetermined wavelength of radiation within the detected radiation.

2. An inspection device as claimed in claim 1 further comprising a collimator positioned between the source and the object to define a beam of radiation directed at the object along said axis.

3. An inspection device as claimed in claim 1 or 2 further comprising a receiving collimator to define an aperture through which scattered radiation is received by the detector.

4. An inspection device as claimed in any preceding claim wherein said detector is comprised of a plurality of sensors configured to form an array such that each sensor measures the intensity for a spectrum of wavelengths of radiation incident upon the sensor.

5. A method of inspecting parcels to detect the presence of selected crystalline materials in the presence of other crystalline and noncrystalline materials comprising:

generating x-ray radiation from a source; conveying an object to be inspected past the source to irradiate the object with the radiation; detecting radiation scattered by the object at a



predetermined angle to detect the presence of a selected crystalline material on or within the object.

6. A method as claimed in claim 5 wherein the selected crystalline material is an explosive or narcotic.

7. A method as claimed in claim 5 or 6 wherein said detecting step comprises measuring the scattered radiation with a plurality of detectors such that each detector measures the radiation scattered by a portion of the object.

8. A method as claimed in claim 7 further comprising:  
generating a plurality of signals from the

radiation measured by each detector;  
forming a spectrum from each of the signals;  
and  
summing the formed spectra to generate a spectra for the object being scanned.

9. A method as claimed in claim 8 further comprising comparing the spectra with a predetermined spectra to determine the presence of selected crystalline materials.

10. A method as claimed in claim 9 wherein the formed spectra from each signal are compared with the predetermined spectra to determine the presence of selected crystalline materials in each portion of the object.

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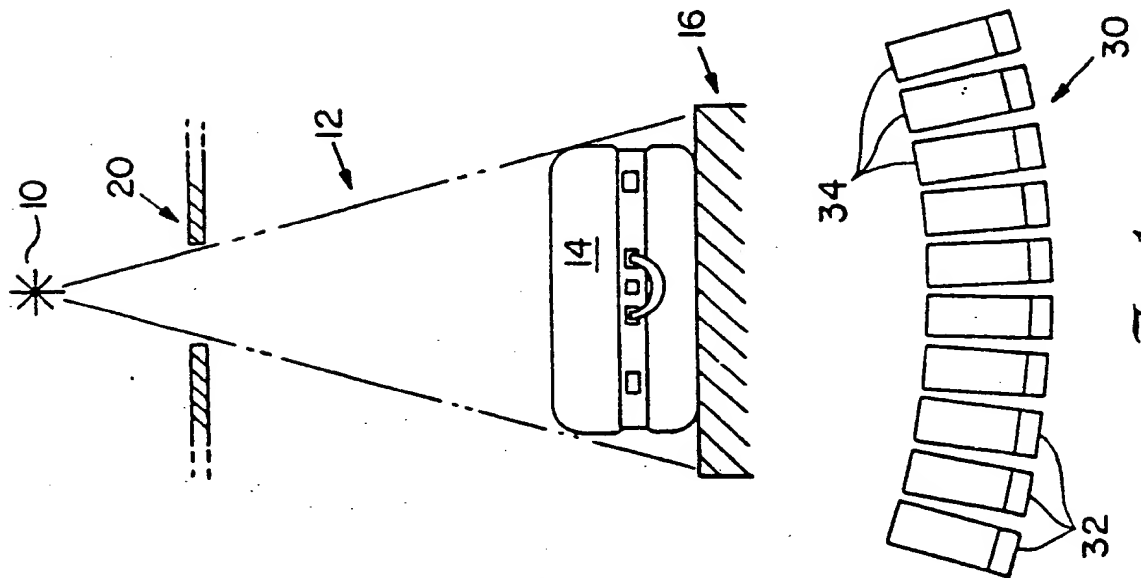
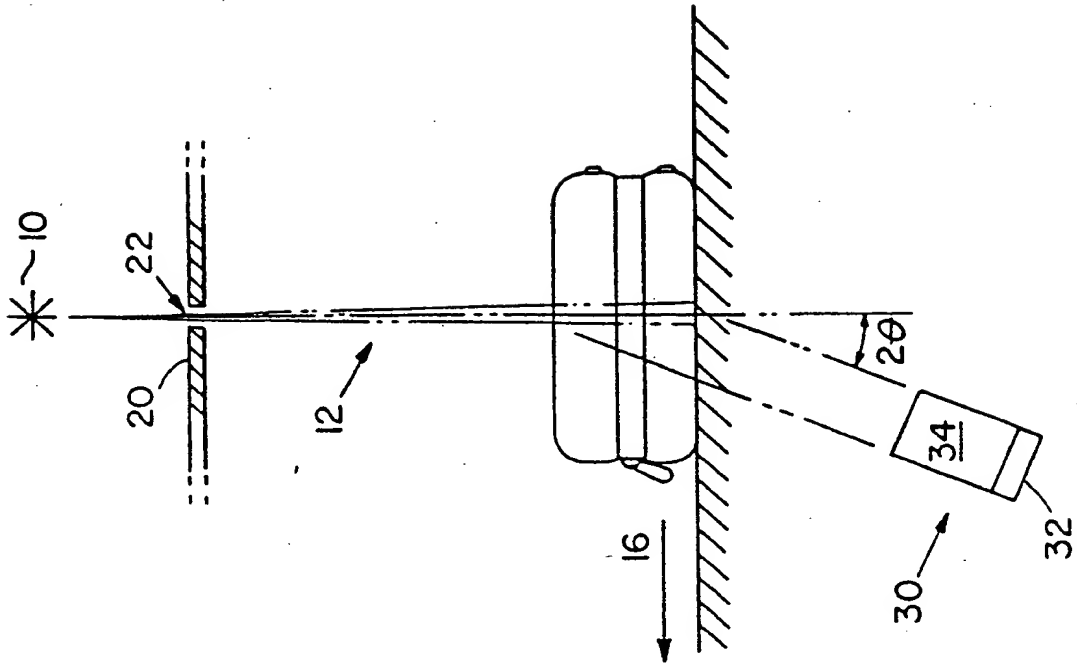


Fig. 3a

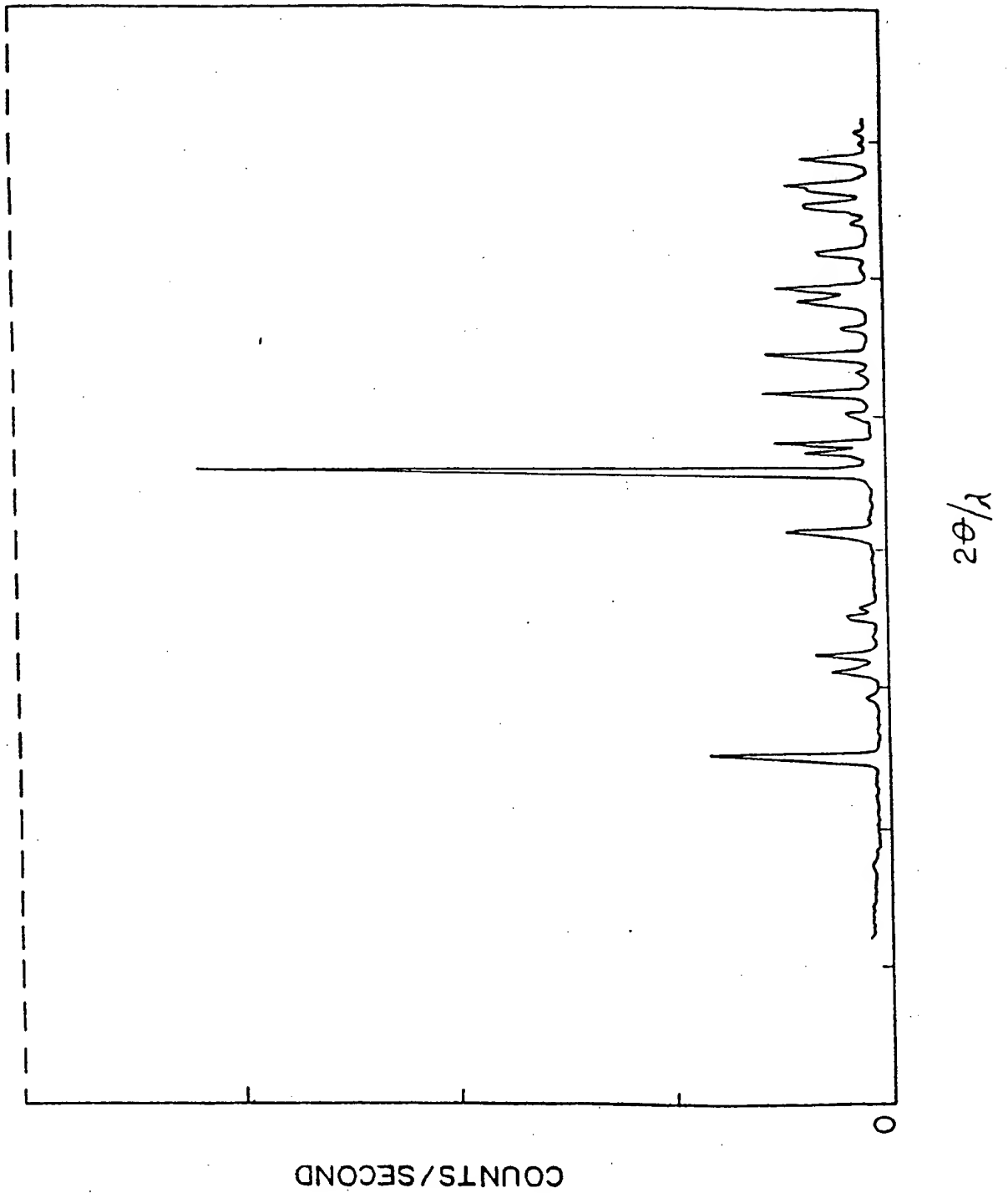


Fig. 4

